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TOUGHNESS AND FLAW RESPONSES IN NONTRANSFORMING CERAMICS: IMPLICATIONS FOR NDE

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INTRODUCTION

Recent developments in the characterization of the strength of ceramics have made it necessary to re-examine several "tradition", long-standing definitions and assumptions that form the modern-day fracture mechanics basis of NDE. Ceramics are very brittle materials. They are highly susceptible to failure from small scale (1-100 μm) "flaws". These flaws may be in the form of machining damage, grain boundary fissures, processing defects (pores or inclusions), etc. Theoretically, flaws have been represented as scaled-down versions of large cracks, so that the macroscopic "laws" of fracture might be assumed to apply at the micro-sale. This philosophy is embodied in the Griffith strength formalism,

$$\sigma_m = T_o/Yc^{1/2} \qquad (1)$$

where c is the flaw size, T_o is the toughness (K_{IC} in metallurgical terminology) and Y is a geometrical constant. Implicit in Eq. 1 are two major conclusions which dictate the entire approach to NDE in ceramics:

(i) Failure occurs spontaneously at the critical stress (σ_m);

(ii) Toughness (T_o) is single-valued.

It is from these two conclusions that the concept of a critical flaw size for failure derives.

In the ceramics literature, the validity of these conclusions and of the extrapolation of large-crack data to the region of microstructure-scale flaws has been questioned by many, but few experimental attempts have been made to answer these questions. Here we shall present some recent data obtained at NBS that seriously questions the entire basis of present-day NDE philosophies for brittle materials. In particular, we shall point out shortcomings in the critical flaw concept due to so-called "crack resistance" (R-curve or T-curve) behavior where toughness becomes a function of crack size [1]. Some potentially beneficial aspects of this behavior will be emphasized.

In setting out to test the validity of Eq. 1 we sought an experimental method which could be used to study systematically a wide range of flaw sizes, from macroscopic crack dimensions down to the scale of the microstructure. The indentation technique [2] was chosen because of its well documented capacity for controlling the scale of the starting flaw. Further, the crack evolution could be directly observed during strength testing in subsequent four-point bend or biaxial flexure (Fig. 1). A detailed fracture mechanics analysis of this test configuration [2,3] allows for the elimination of crack size in favor of indentation load, P, from Eq. 1, retaining the assumption of a single-valued toughness, T_o:

Hence by observing the $\sigma_m(P)$ response, we can test the basis for macroscopic to microscopic extrapolations; if T_o is indeed a single-valued constant, σ_m should plot as a straight line with slope -1/3 in logarithmic coordinates.

Alumina was chosen as the "model" ceramic for the experimental study because of its availability in a wide range of microstructures. We show results here for single crystal sapphire and two polycrystalline materials, one nominally pure and the other containing a glassy grain boundary phase.

The results are shown in Fig. 2. Each data point represents the mean of about 10 specimens at a given load; error bars are omitted, but standard deviations are typically 10%. The curves through the data are best fits [4,5]. The linear fits for sapphire and the glassy polycrystalline material are in accord with Eq. 2, suggesting that the macroscopic toughness can indeed be extrapolated back to the flaw scale. However, the fit for "pure" alumina deviates dramatically from the ideal linear behavior at smaller flaw sizes. Thus, for this, third material predictions based on extrapolations from the macroscale greatly overestimate the actual strengths. It is as though the toughness, T, is systematically diminished as the flaw size gets smaller, i.e., consistent with R-curve (T-curve) behavior. On the other hand, we have the desirable feature of a region of "flaw tolerance" where the strength is constant over a range of flaw sizes.

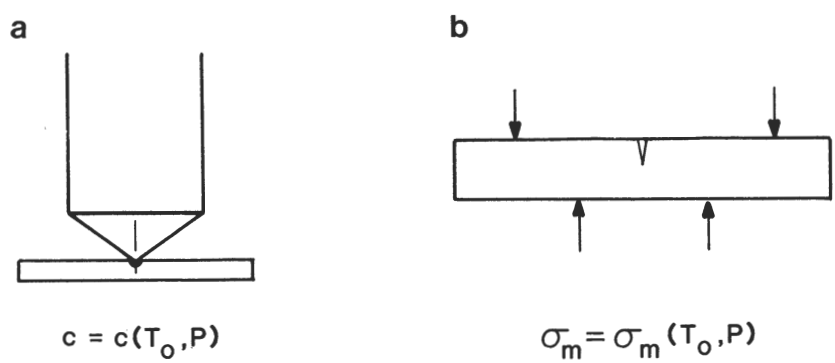

Figure 1. Schematic of indentation flaw test used to observe crack evolution to failure: (a) indentation, to introduce controlled flaw; (b) bend test, to measure strength of specimen with flaw.

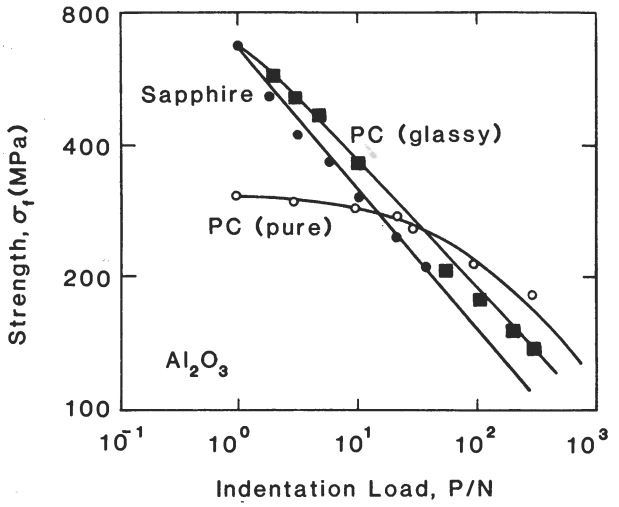

Figure 2. Inert strength vs indentation load of three aluminas, single crystal (sapphire) and polycrystalline with and without glassy grain boundary phase (PC-glassy and PC-pure, respectively).

From these results, we conclude:

(i) Extrapolation of macroscopic fracture data unconditionally into small-flaw regions can be dangerous;

(ii) Toughness is not generally single valued, but can be a function of crack size, T(c);

(iii) The toughness function (T-curve) is microstructure sensitive, and the grain boundary structure appears to hold the key to this sensitivity.

MECHANISMS OF T-CURVE BEHAVIOR

There are several possible mechanisms which have been put forward to explain T-curve behavior. The most popular of these are the "frontal zone" mechanisms. Martensitic phase transformation is probably the most powerful of all the toughening mechanisms but, to date, has been observed exclusively in zirconias [6]. The concept of microcracking has also received much attention [7] in nontransforming ceramics. In both these mechanisms, there is a frontal zone which travels with the extending crack tip and thereby dissipates energy from the loading system. Somewhat remarkably, very little attempt has been made to verify these (or indeed any other) mechanisms by direct observation (except in the transforming zirconias).

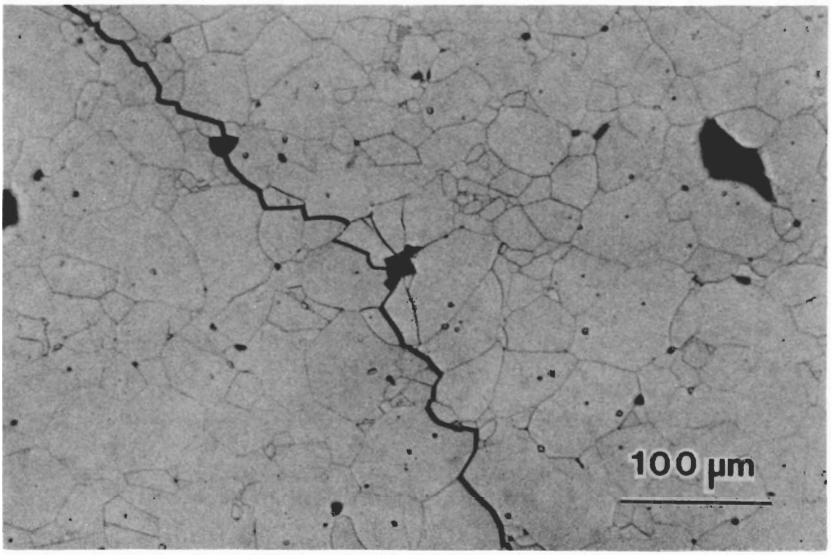

Figure 3. Vickers indentation (P = 5N) site of "fractured" alumina disc. Specimen thermally etched to reveal grain structure.

IN SITU OBSERVATIONS OF CRACK EVOLUTION IN ALUMINA

By using the arrangement in Fig. 1 and focussing a microscope onto the indentation site during stressing, the crack evolution to failure could be observed directly [8]. These observations led to some surprises. Whereas in sapphire failure was spontaneous, in the "pure" polycrystalline alumina it certainly was not. The cracks in the latter material were stabilized. At "failure" these specimens seemed to be fully fractured (the crack ran from edge to edge and through the thickness of the sample) yet remained intact.

The center region of a broken specimen of the latter material is shown in Fig. 3. At initial loading the indentation remained stationary, confined at surrounding grain boundaries until at a critical point the cracks suddenly "popped in". With subsequent load increments, grain-dimension "jumps" occurred in a stable but erratic manner for several millimeters before failure. Despite intensive searching, no evidence could be found for any frontal zone mechanism, microcracking or otherwise. On the other hand, inspection of the crack interface behind the tip revealed a high density of "active" regions where grains remained attached to both walls. The crack tip was clearly held up by these partially attached grains. Two specific examples of active grain sites are shown in Fig. 4. In both cases in Fig. 4, secondary grain fracture is evident, suggesting that the interfacial restraining forces must be high.

To summarize the experimental observations:

(i) Crack growth in the "pure" alumina was discontinuous over small groups of grains, yet stable over 10-100 grains (cf. relatively spontaneous fracture in the other aluminas in Fig. 2);

(ii) Grain attachment sites were active behind the crack tip, over many millimeters in the "pure" alumina;

(iii) No evidence was found for a frontal microcrack zone.

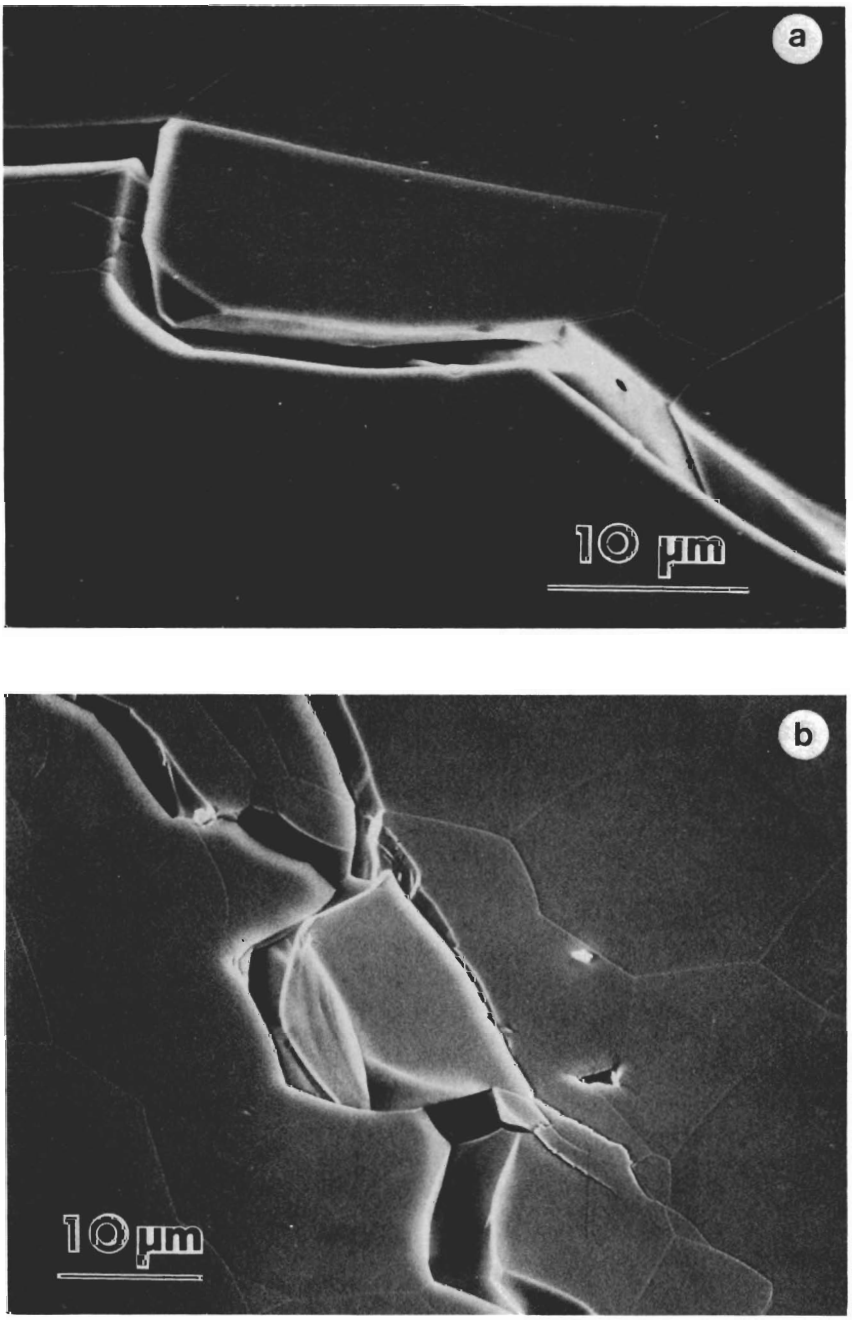

Figure 4. Scanning electron micrographs showing two examples of secondary microfracture about bridging grains.

Thus the macroscopic evidence implies that the T-curve behavior in our "pure" alumina is due primarily to bridging forces at the crack interface. More recent wo~k by Swanson on other materials [9] suggests that this observation may generally be true of other ceramic types as well.

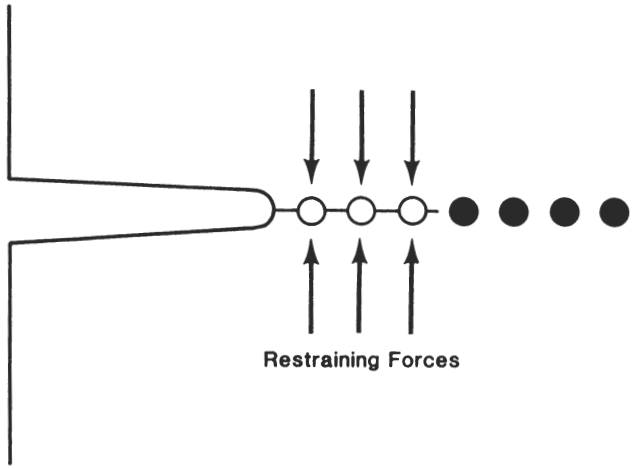

Figure 5. Schematic of bridging model (shown here for crack growth from notch).

MODEL

A fracture mechanics model of the bridging mechanism has been developed [10] (see Fig. 5). Circles denote bridging sites; open and closed circles distinguish active sites behind the tip and future sites ahead of the tip. The sequence of calculations involved is as follows:

(i) The T-curve is taken to be expressible as the sum of the intrinsic (grain boundary) toughness, T_o, and an "internal" restraining term, K_i, i.e.

$$T(c) = T_o - K_i(c) \qquad (3)$$

For restraining forces, K_i is negative;

(ii) The internal closure force, $K_i(c)$, is determined by integrating the closure forces over the bridging zone, assuming a specific force-separation law. Since more bridges are intersected as the crack grows, K_i is an arithmetically increasing function of c;

(iii) The critical condition for crack instability, $K_a(c) = Y \sigma_a c^{1/2} = T(c)$, $dK_a/dc = dT/dc$, [11] is computed to determine the strength vs load function, $\sigma_m(P)$;

(iv) From the $\sigma_m(P)$ data in Fig. 2, the T(c) function is (numerically) deconvoluted.

Figure 6 is a composite plot of the results for those aluminas in Fig. 2 using this approach (with several approximations in the analysis). Some of the more important features to note are:

(i) The crack size scale of the T-curve can be large, of order millimeters, consistent with the scale of the observed bridging zones;

(ii) The T-curves are microstructure sensitive: the only difference between the aluminas represented in Fig. 6 are the grain sizes and grain boundary phases;

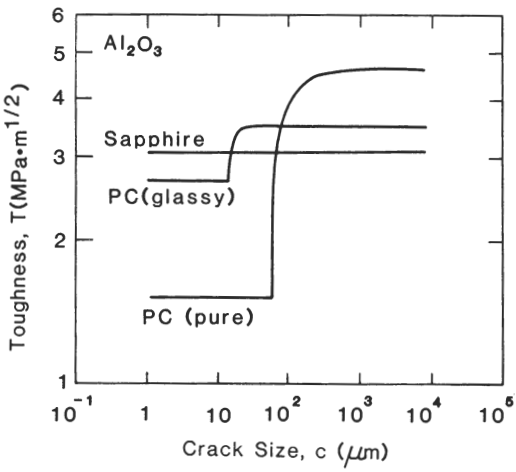

Figure 6. Toughness as a function of crack length deconvoluted for aluminas from Fig. 2.

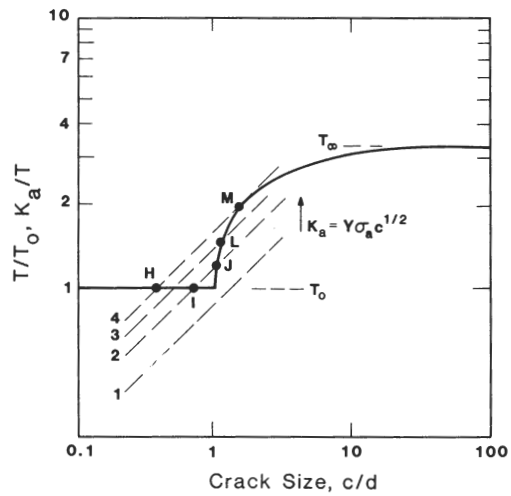

Figure 7. T-curve construction.

(iii) There appears to be an inverse relationship between T_o and T_∞ (lower and upper plateau levels): thus large-scale toughness can be a poor indicator of small-scale toughness.

Consider now how the results in Fig. 6 explain the observed crack evolution for materials with strong T-curve behavior (e.g., the polycrystalline "pure" material in Fig. 6). A construction for such a material is reproduced in schematic form in Fig. 7. The solid curve represents the $T_{(c)}$ function and lines 1 through 4 represent $K_a = Y \sigma_a c^{1/2}$ "loading lines" for successively increasing values of applied stress, σ_a Suppose our flaw has initial size corresponding to point I. Then the crack remains stationary until stage 2 is reached in loading, at which point abrupt pop-in occurs, along IJ. With further loading the crack subsequently progresses stably through JLM along the curve, until at stage 4, failure ensues. Actually, a more exact numerical deconvolution than we have attempted in our data analysis would yield several secondary plateaus along the rising T-curves in Fig. 6, consistent with the observation that crack growth occurs in discrete jumps throughout its evolution. Thus the failure stress is determined uniquely by the tangency condition at M, independent of the initial crack size.

IMPLICATIONS

What are the implications of our results concerning NDE in ceramics? First, we have shown that materials which exhibit strong T-curve behavior can be extremely "flaw tolerant". The failure stress for these materials is independent of the initial flaw size. For the engineer, this is an extremely attractive prospect, for not only is the concept of a well-defined design stress feasible, but the material is now much less susceptible to strength degradation in service. However, at the same time this raises the question as to whether we can retain the notion of critical flaw size as a basis for screening. Secondly, there can be strongly enhanced crack stability in these materials; the cracks can grow large distances (tens or hundreds of grains) over the rising portion of the T-curve, prior to failure. Importantly, this growth can occur discontinuously. Thus, the precursor growth stage may be usefully employed as an "early warning" of impending failure. The possibilities of turning this to advantage are clear.

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